भारतीय मानक
Indian Standard

IS 17200 : 2019

वैलिडामाईसिन, तकनीकी — विशिष्टि

Validamycin, Technical — Specification

ICS 65.100.30

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Pesticides Sectional Committee, FAD 01

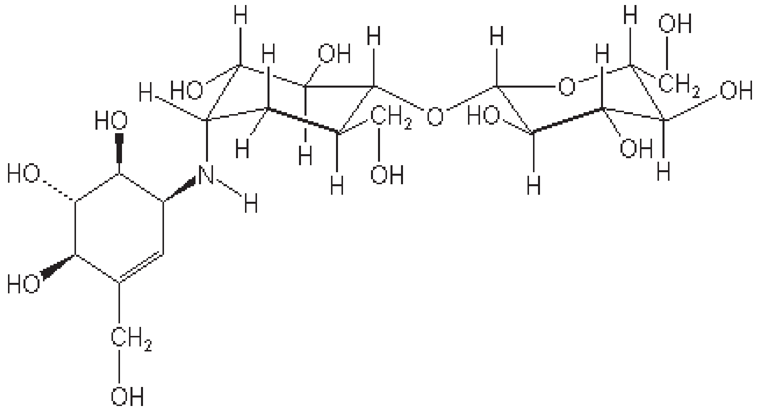
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Validamycin technical is employed in the preparation of fungicidal formulations.

IUPAC name for validamycin is (2R,3R,4S,5S,6R)-2-[(1R,2R,3S,4S,6R)-2,3-dihydroxy-6-(hydroxymethyl)-4-[[(1S,4R,5S,6S)-4,5,6-trihydroxy-3-(hydroxymethyl)cyclohex-2-en-1-yl]amino]cyclohexyl]oxy-6-(hydroxymethyl)oxane-3,4,5-triol.

The empirical and structural formula and molecular mass of validamycin are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{20}H_{35}NO_{13}$		497.49

In the formulation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and *Rules*, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

VALIDAMYCIN, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for Validamycin, technical.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6940 :1982	Method of tests for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirements for packaging of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a brownish grey crystalline powder free from extraneous impurities or added modified agents.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in fibre board drums provided internally with low density polythene lining of thickness not less than 0.062 mm. The drums shall have mild steel jacketed circular rim on top only. The general requirements given in IS 8190 (Part 1) shall be followed.

5 MARKING

5.1 The sack shall bear legibly and indelibly the following information in addition to any other information as is necessary under *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal Validamycin content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules,* 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Validamycin, Technical

(*Clause* 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Validamycin content, percent by mass, *Min*	64	A	—
ii)	Moisture content, Percent by mass, *Max*	2.0	—	**4**

6 SAMPLING

Representative samples shall be drawn as prescribed in IS 10946.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col **4** and **5** of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF VALIDAMYCIN CONTENT

A-1 PRINCIPLE

Validamycin content is determined by the high performance liquid chromatograph (HPLC) equipped with UV-VIS detector by using an internal standard technique.

A-2 APPARATUS

A-2.1 The high performance liquid chromatograph (HPLC) equipped with UV-VIS detector and coupled to a printer-plotter-cum-integrator. The operative condition suggested below are typical which can be changed provided the standardization is done:

Column : 250 mm × 4.6 mm, 5 micron, SS packed with C-18
Detector : UV (220 nm)
Mobile phase : Dissolve 47 mg sodium borate ($Na_2B_4O_7.10H_2O$), 100 mg potassium dihydrogen phosphate (KH_2PO_4) and 40 ml acetonitrile into a 1 000 ml volumetric flask, make up to the mark with HPLC grade water
Flow rate : 1.5 ml/min
Injection volume : 20 µl
Temperature : Ambient

A-2.2 Microliter Syringe — 50 µl capacity.

A-2.3 Analytical Balance

A-2.4 Standard Glassware

A-3 REAGENTS

A-3.1 Validamycin Standard, of known purity.

A-3.2 Internal Standard-Inosine — AR or equivalent grade.

A-3.3 Water — HPLC Grade.

A-3.4 Acetonitrile — HPLC grade.

A-3.5 $Na_2B_4O_7.10H_2O$ — AR or equivalent grade.

A-3.6 KH_2PO_4 — AR or equivalent grade.

A-4 PROCEDURE

A-4.1 Solvent System

Dissolve 47 mg sodium borate ($Na_2B_4O_7.10H_2O$), 100 mg potassium dihydrogen phosphate (KH_2PO_4) and 40 ml acetonitrile into a 1 000 ml volumetric flask, make up to the mark with HPLC grade water.

A-4.2 Preparation of Internal Standard

Weigh accurately 0.015 g of inosine into a 100 ml volumetric flask, make up to the mark with HPLC grade water.

A-4.3 Preparation of Reference Standard Solution

Weigh accurately 0.03 g of validamycin standard into the 100 ml volumetric flask, make upto the mark with water and take 5 ml sample from this solution into 25 ml volumetric flask, add 5 ml internal standard solution make up to the mark with HPLC grade water.

A-4.4 Preparation of Sample Solution

Weigh accurately a sample containing equivalent to 0.03 g of Validamycin and proceed as given in **A-4.3**.

A-4.5 Estimation

Introduce separately 20 µl each of the final solution of standard and sample into HPLC unit and adjust the attenuation in such a way that Validamycin and internal standard peaks are obtained within the scale.

A-5 CALCULATION

Validamycin content, percent by mass =

$$\frac{M_1 \times A_1 \times A_3}{M_2 \times A_2 \times A_4} \times P$$

Where,

M_1 = Mass of Validamycin standard;
M_2 = Mass of Validamycin sample;
A_1 = Peak area of Validamycin, in sample solution;
A_2 = Peak area of Validamycin, in standard solution;
A_3 = Peak area of internal standard, in standard solution;
A_4 = Peak area of internal standard, in sample solution; and
P = Percent purity of standard Validamycin.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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